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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to 1 \*\*\*\* solvent form moisture hardening mold urethane system liquefied type adhesives.

[0002]

[Description of the Prior Art] When the urethane prepolymer (henceforth a "prepolymer") which has an isocyanate radical at the end is used as a principal component, and the isocyanate radical reacts with the moisture in air, and the moisture in adherend and carries out hardening bridge formation, 1 liquid moisture hardening mold urethane system adhesives form an adhesives layer, and paste up various adherends.

[0003] Conventionally, the moisture hardening mold urethane system adhesives of 1 liquid made polyols and the superfluous poly isocyanates react without using the bottom of existence of an organic solvent, or this, compounded the urethane prepolymer which has an isocyanate radical at the end, blended the filler, the organic solvent, the thixotropy agent, the curing catalyst, etc. with this if needed, and were using them as adhesives.

[0004] If it is a solvent form among 1 liquid moisture hardening mold urethane system adhesives, in order to dilute using a lot of volatile organic solvents to a solvent, molecular weight of an urethane prepolymer may be enlarged extremely. Therefore, the initial adhesive property which an organic solvent evaporates and is discovered immediately can give the initial adhesion condition (henceforth an "initial tuck") of having excelled with the property of a macromolecule polymer. As a property of this solvent form, in order that an organic solvent may help wettability required for the adhesion which is the difficulty of a macromolecule polymer by both humid effectiveness and the dilution effect, an adhesive property is also still better.

[0005] In order that the organic solvent used for these 1 liquid solvent form moisture hardening mold urethane system adhesives may obtain the manifestation strength of an initial tuck at an early stage, the so-called medium boiler whose boiling points, such as the so-called low boiler 100 degrees C or less or toluene, a xylene, and methyl isobutyl ketone, the boiling point represented by ethyl acetate, a methyl ethyl ketone, and the methylene chloride is 100 degrees C - 150 degrees C is used general-purpose. Since desiccation is delayed, a high boiler, a plasticizer, etc. are extent used together auxiliary if needed.

[0006] law, such as Fire Service Law since the effect which exerts these solvents on the odor by the vaporization of the solvent at the time of spreading or an operator's health is a big problem and it is the inflammable dangerous substance, when the cautions to fire prevention become important, — it is set as the object of regulation and a limit is received also in storage quantity.

[0007] Thus, although 1 liquid solvent form moisture hardening mold urethane system adhesives possess the speed of a manifestation and strength, and a good adhesive property, it is common knowledge that the risk based on an organic solvent and a harmful property serve as a serious difficulty of these adhesives in society today. [an initial tuck]

[0008] a means to cancel the difficulty ---less -- a solvent -- although it is-izing, it does not realize only by making an organic solvent into zero. That is, a technical difficulty that the adhesive property which spreading is difficult in the first place when it is required for the urethane prepolymer usually used for 1 liquid moisture hardening mold urethane system adhesives although it is called for that a first stage tuck advanced with adhesives is discovered at an early stage as much as possible to be liquefied and the prepolymer of all possible large molecular weight in the range is used, and is equal to a solvent form the second since a macromolecule polymer is lacking in wettability is not acquired at all will be produced. [0009] Since the prepolymer of low molecular weight must be used for 1 \*\*\*\* solvent form moisture hardening mold urethane system adhesives actually marketed conventionally, a difficulty that it is deficient in an initial tuck and a manifestation is slow is pointed out. Furthermore, in order to give spreading workability, it is the actual condition which must be diluted a little with that organic solvent. Moreover, in an adhesive property, since it is lacking in wettability compared with a solvent form, the present condition is that both adhesive property and bonding strength are inferior to charges of a difficulty binder, such as plastics.

[0010]

[Problem(s) to be Solved by the Invention] This invention improves the spreading workability which is the difficulty of the conventional 1 \*\*\*\* solvent form moisture hardening mold urethane system adhesives, makes the outstanding adhesive property give, and is to enable the early manifestation of a good initial tuck based on fast curability.

[0011]

[Means for Solving the Problem] this invention person etc. was looking for the diluent which was excellent in the reducibility replaced with the conventional low-boiling point - an inside boiling point organic solvent to the moisture hardening mold adhesives which used the urethane prepolymer. Consequently, although the N-methyl-2-pyrrolidone was found out, according to literature search, it got to know that this diluent was used as a solvent at the time of an urethane polymerization with dimethylformamide. However, this diluent discovered giving not only the mere dilution effect but various unexpected properties.

[0012] That is, discovery of giving the adhesive property which was excellent to charges of a difficulty binder, such as not only leading this diluent to hypoviscosity to a prepolymer but a metal, rubber, plastics, etc., and effectiveness which is not known at all, such as a certain thing, became the beginning of this invention to the facilitatory effect of a cure rate further.

[0013] In addition, the hypoviscosity as used in the field of this invention is the field of the viscosity which becomes liquefied at a room temperature, and, specifically, the viscosity (23 degrees C) in rotational frequency 20 r/min of B mold rotational viscometer shows 100 or less Pa-s.

[0014] Furthermore, when the screw (morpholino ethyl) ether which is a specific urethane curing catalyst, the screw (2, 6-dimethyl morpholino ethyl) ether, and the screw (3, 5-dimethyl morpholino ethyl) ether were used together, it discovered that the facilitatory effect of a cure rate was remarkable by the interaction, maintaining the above-mentioned property. Therefore, a cure rate becomes quick from the case where this specific urethane curing catalyst is used independently.

[0015] When research is further continued aiming at reduction-izing of prepolymer viscosity, the balance of an adhesion function, or improvement in an initial tuck, then, to hypoviscosity-izing of prepolymer viscosity, and the balance of an adhesion function For the early manifestation of the initial tuck discovered as a technical means to use together specific polyether polyol and specific polyester polyol, fast curability, and its result Discovered the technical means using a specific amine compound as a curing catalyst, it was made to reach to the further amelioration of the purpose of this invention, and this invention was completed.

[0016] Hereafter, the technical means of this invention is expressed concretely. That is, invention of claim 1 is 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent which can be applied at the room temperature characterized by having blended the N-methyl-2-pyrrolidone and considering as a high adhesive property by hypoviscosity to the urethane prepolymer of an isocyanate end.

[0017] The rate that invention of claim 2 blends a N-methyl-2-pyrrolidone to the urethane prepolymer of an isocyanate end is 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent which can be applied at the room temperature in the means of claim 1 characterized by considering as a high adhesive property by the hypoviscosity made into 0.1 - 30 mass section to the prepolymer 100 mass section.

[0018] It is 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent which can be applied at the room temperature in the means of claim 1 characterized by invention of claim 3 improving the balance of adhesive ability by the hypoviscosity which is the urethane prepolymer of the isocyanate end obtained by the polyester polyols from which the urethane prepolymer of an isocyanate end was obtained by the reaction of polyoxypropylene polyol, and a polyhydric alcohol and a multiple-valued carboxylic acid, and the poly isocyanate, or claim 2.

[0019] As opposed to 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent [ in / in invention of claim 4 / the means of either claim 1 thru/or claim 3 ] The screw (morpholino ethyl) ether, the screw (2, 6-dimethyl morpholino ethyl) ether, It is 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent which can be applied at the room temperature characterized by improving the fast curability which comes to blend one or more sorts of amine compounds chosen from the screw (3, 5-dimethyl morpholino ethyl) ether.

[0020] With the polyol which is the start raw material of the urethane prepolymer of this invention, it can classify to polyether polyol and polyester polyol.

[0021] as polyether polyol -- polyoxyalkylene diol (concrete -- polyoxy ethylene glycol --) A polyoxypropylene glycol, polyoxy butylene glycols, etc. and these copolymers, polyoxyalkylene triol (concrete -- ethylene oxide and propylene oxide --) Polyoxyalkylene polyols, such as what carried out the addition polymerization of a glycerol, the trimethylol propane, etc. to butylene oxide, styrene oxide, etc., In addition, the diol to which ethylene oxide or propylene oxide was made to add by using a bisphenol, sugars, amines, etc. as a start raw material, Polyether polyol, the polyoxy tetramethyl glycol obtained by the ring opening polymerization of a tetrahydrofuran can be mentioned.

[0022] Since liquefaction is easy at this inner hypoviscosity, use of polyoxyalkylene diol and polyoxyalkylene triol is desirable.

[0023] As long as it is the compound which has a hydroxyl group at the end which repeats an ester bond and it has as a unit as polyester polyol, a well-known thing can be used widely conventionally. concrete -- a multiple-valued carboxylic acid (mainly an adipic acid and a phthalic acid) and polyhydric alcohol (concrete -- a glycol --) By the ring opening polymerization of the condensed system polyester polyol; epsilon-caprolactone obtained by dehydration condensation with triol etc. Lactone system polyester polyol obtained; by phosgene-izing of polyol, or the ester exchange reaction by diphenylene carbonate Polyester polyol by the polyaddition reaction of the polycarbonate diol; polycarboxylic acid anhydride and diepoxide which are obtained; urethane denaturation polyester polyol which introduced the urethane group etc. into the interior of a molecule can be mentioned.

[0024] Since the adhesive property which was excellent in this is given, use of the condensed system polyester polyol obtained by the condensation-ized reaction of polyhydric alcohol, such as ethylene glycol, a diethylene glycol, propylene glycol, 1,4-butanediol, 1,6-hexanediol, and neopentyl glycol, and multiple-valued carboxylic acids, such as an adipic acid, sebacic acid, isophthalic acid, and trimellitic acid, is desirable.

[0025] As polyols other than the above, ethylene glycol, propylene glycol, Dihydric alcohol, such as 1,4-butanediol, a glycerol, trimethylol propane, polyhydric alcohol, such as neopentyl glycol, and hydroxyl-group end polybutadiene (1 and 2-adduct --) 1, 4-adduct, the so-called rosin denaturation polyol that has a rosin frame in intramolecular, The amine polyol which has amine structure in intramolecular, and the so-called silicon system diols which introduced the carbinol into the both ends of the polysiloxane called a carbinol denaturation silicone oil The so-called fluororesin system polyol which has a hydroxyl group for the so-called acrylic polyols which made acrylic ester (methacrylic ester is also included) the frame, and a frame at the end of a fluororesin (for example, copolymerization resin of 2 fluoride ethylene and acrylic) can be mentioned.

[0026] As poly isocyanate used by this invention, the poly isocyanate compounds, such as two or more aliphatic series which has an average of 2-3 isocyanate radicals preferably, aromatic series, and an alicycle group, are used into a molecule.

[0027] Specifically For example, 2, 4-toluene diisocyanate (henceforth "2, 4-TDI"), 2,6-toluene diisocyanate (it is hereafter called "2, 6-TDI".) Isomer mixture [ of 2, 4-TDI, and 2 and 6-TDI ], 4, and 4'-diphenylmethane diisocyanate (it is hereafter called "4 and 4'-MDI".) 2, 4'-diphenylmethane diisocyanate (it is hereafter called "2 and 4'-MDI".) isomer mixture (trade name: -- RUPURANETO MI --) with 4 and 4 '-MDI, 2, 4'-MDI The BASF Japan, Ltd. make, the carbodiimide denaturation MDI, polymethylene polyphenyl polyisocyanate (it is hereafter called a "polymeric MDI".) Hexamethylene diisocyanate (it is hereafter called "HMDI".) Xylenediisocyanate (it is hereafter called "XDI".) Metaxylenediisocyanate, 1,5-naphthalene diisocyanate, Buret poly isocyanate compounds, such as isocyanate compound; Sumi Joule N (the Sumitomo Bayer urethane company make), such as Hydrogenation MDI, Hydrogenation XDI, and isophorone diisocyanate; Desmodurs IL and HL (Bayer A.G. company make), The poly isocyanate compound which has isocyanate rings, such as Coronate EH (Japanese polyurethane industrial company make), Sumi Joule L (the Sumitomo Bayer urethane company make) Adduct poly isocyanate compounds, such as Coronate HL (Japanese polyurethane industrial company make), etc. can be mentioned.

[0028] Among this, it is desirable in the strength of an initial tuck to use mixture and these crude materials with 4 and 4'-MDI and these crude-materials, 4, and 4 '-MDI, 2, 4'-MDI in respect of an adhesive property, bonding strength, and good spreading.

[0029] To the mixed polyol usually obtained with one sort or two sorts or more of combination chosen from polyether polyol, polyester polyol, or the other polyols, the urethane prepolymer of an isocyanate end makes the superfluous poly isocyanate act, and is compounded.

[0030] From the chemical equivalent of the hydroxyl group of all polyol components, the superfluous poly isocyanate here means that the chemical equivalent of an isocyanate radical is superfluous, and can express the relation of the chemical equivalent with a NCO/OH ratio. That is, since this invention is characterized by the liquefied thing, 1.5 or more usually have a NCO/OH ratio desirable [ if a NCO/OH ratio exceeds 1 theoretically, it can become an isocyanate end, but / this invention ]. Although especially the upper limit of a NCO/OH ratio is not limited, consideration, then its NCO/OH ratio 10.0 less or equals are desirable in evils, such as an increment in foaming in the middle of hardening delay and moisture hardening. It is especially liquefied, and in order to lead to the prepolymer of hypoviscosity, while taking into consideration the class of polyol, the number of functional groups, molecular weight, etc., especially the thing for which a NCO/OH ratio is adjusted about to 1.5 to 8.0 is desirable. [0031] Although polymerization temperature and especially polymerization time amount are not restricted, either, it is [ after mixing polyol and the poly isocyanate ] usually good under a nitrogen gas air current to make it react at 50-100 degrees C for 3 to 8 hours.

[0032] Although the N-methyl-2-pyrrolidone used for this invention may blend a commercial item as it is, it is more desirable for commercial dehydrating agents, such as a molecular sieve and silica gel, to remove moisture, in order to acquire long-term storage stability.

[0033] The loadings of the N-methyl-2-pyrrolidone used for this invention have desirable 0.1 - 30 mass section to the prepolymer 100 mass section. If fewer than the 0.1 mass section, adhesives will be turned hypoviscosity up, the adhesive property over the charge of a difficulty binder will be given, when it is scarce to acquire the effectiveness of cure-rate promotion further and it exceeds 30 mass sections, the manifestation effectiveness of an initial tuck fades conversely and the effectiveness which a N-methyl-2-pyrrolidone originally has stops being discovered -- reservation of the bonding strength for moving to the next activity becomes difficult. The most desirable range of the loadings of the N-methyl-2-pyrrolidone which demonstrates the above-mentioned effectiveness most notably is 0.5 - 30 mass section.

[0034] When blending with the adhesives of this invention, you may blend at which stage in the synthetic process of an urethane prepolymer, and the production process of adhesives, but since a N-methyl-2-pyrrolidone tends to absorb water, it is more desirable among the production process of

adhesives with little effect of moisture. It is blending in the last production process of adhesives most preferably.

[0035] Among the polyoxypropylene polyols in this invention, polyoxypropylene diol carries out the addition polymerization of the propylene oxide by making propylene glycol, neopentyl glycol, etc. into an initiator, and is compounded. Moreover, polyoxypropylene triol carries out the addition polymerization of the propylene oxide by making a glycerol and trimethylol propane into an initiator, and is compounded. As for these, the thing of 400-20000 is usually used for molecular weight. [0036] The polyester polyols (trade name: ADEKA new ace F15-22, molecular weight 2000, two organic functions, Asahi Denka Kogyo K.K. make, etc.) obtained by the condensation reaction of the polyester polyols (trade name: ADEKA new ace F7-68, the molecular weight 1000, two organic functions, Asahi Denka Kogyo K.K. make, etc.) and the diethylene glycol which are specifically [ the polyester polyols obtained by the reaction of the polyhydric alcohol and the multiple-valued carboxylic acid in this invention ] obtained by the condensation reaction of propylene glycol and an adipic acid, and an adipic acid are mentioned.

[0037] It becomes the screw (morpholino ethyl) ether of this invention, the screw (2, 6-dimethyl morpholino ethyl) ether, and the screw (3, 5-dimethyl morpholino ethyl) ether from the general formula of the following chemical formula 1.

[0038]

[Formula 1]
$$(R^2)_n$$

$$N-CH_2-CH_2-O-CH_2-CH_2-NO$$

[0039] The inside of a formula, and R2 C1 - C3 This compound that an alkyl group and n show the integer of 0-4, and is expressed with this formula is an urethane curing catalyst.

[0040] Since storage stability is excellent, it is called the so-called bubble type amine system catalyst which promotes the reaction of the suitable isocyanate radical and suitable moisture for manufacture of urethane foam and not only manufacture of urethane foam but storage stability is excellent, this screw (morpholino ethyl) ether is a general catalyst for urethane polymerizations currently used for many applications, such as caulking materials, such as an urethane sealant.

[0041] The blending ratio of coal of this urethane curing catalyst is 0.05 - 10 mass section to the urethane prepolymer 100 mass section. If [ than 10 mass sections ] more [ the hardening promotion operation by the interaction with a N-methyl-2-pyrrolidone is scarce when fewer than the 0.05 mass section and ], storage stability, such as a with-time viscosity rise of adhesives, will get extremely bad. [0042] You may use together suitably [ for the purpose of organic metal system urethane catalysts, such as this curing catalyst, other amine system urethane catalysts, and a dibutyl tin JIRAU rate, ]. [0043] The production process of the adhesives of this invention is indicated below. At a production process, it manufactures to the well-closed container which intercepted moisture by blending various modifiers, such as an inorganic system filler, a thixotropy agent, a plasticizer, a diluent, a tackifier, an adhesive grant agent, a curing catalyst, a moisture binder, a stabilizer, a coloring agent, and an organic solvent, and carrying out mixed processing to a prepolymer, under a desiccation nitrogen air current. [0044] Since these adhesives are destabilized under the effect of moisture at the time of a production process and storage, as for the raw material which has moisture by the feed ingredient, it is desirable to remove moisture as much as possible, when acquiring the stability of the quality over the long period of time of adhesives.

[0045] as an inorganic system filler used for this invention, inorganic system fillers which can be industrially used as a filler, such as a calcium carbonate, clay, talc, magnesium oxide, diatomaceous earth, and glass balun, mention -- having -- respectively -- independence -- or it can be mixed and used. [0046] A silica, hardening castor oil, etc. are mentioned as a thixotropy agent after superfines. [0047] as a plasticizer, dioctyl phthalate, dibutyl phthalate, dilauryl phthalate, butyl benzyl phthalate, dioctyl adipate, di-isodecyl adipate, trioctylphosphate, a mineral spirit, etc. mention, for example --

having -- respectively -- independence -- or it can be mixed and used.

[0048] As a diluent, ester, such as a petroleum system hydrocarbon diluent, 3-methyl-3-methoxy butyl acetate, and glutaric-acid dimethyl, is mentioned.

[0049] As a tackifier, a well-known thing can be used conventionally. Specifically with a natural resin system tackifier, there is terpene system resin, such as rosin system resin; terpene resin, such as rosin, denaturation rosin, and rosin ester, aromatic series denaturation terpene resin, hydrogenation terpene resin, and terpene phenol resin. Moreover, in a synthetic-resin system tackifier, there is phenol system resin; xylene resin, such as pure monomer system petroleum resin; alkylphenol resin, such as petroleum resin; coumarone-indene resin; styrene resin, permutation styrene resin, etc., such as aliphatic series system (C5 system) petroleum resin, aromatic series system (C9 system) petroleum resin, copolymerization system (C5/C9 system) petroleum resin, hydrogenation petroleum resin, and DCPD system petroleum resin, and rosin denaturation phenol resin, etc.

[0050] Among this, a liquefied tackifier and the tackifier which can dissolve in an urethane prepolymer in a solid can be used in ordinary temperature, and softening temperature is a thing 130 degrees C or less preferably in hard resin.

[0051] As an adhesive grant agent, silane coupling agents (gamma-aminopropyl trimethoxysilane, gamma-glycidoxy propyltriethoxysilane, reactant of both this compound, etc.) etc. are mentioned. [0052] Tosyl isocyanate, a zeolite, etc. are mentioned as a moisture binder.

[0053] As a stabilizer, azo compounds, such as ultraviolet ray absorbents, such as tinuvin 326 and 2 and 4-dihydroxy benzophenone, a metal chelate like Zn and nickel-dibutyl dithio carbamate, p-hydroxyazobenzene, and p-aminoazobenzene, etc. are mentioned, and it is used for the purpose of optical stabilization.

[0054] As a coloring agent, organic dye, such as inorganic system coloring agents, such as titanium oxide, ferrous oxide, chromic oxide, a cadmium sulfate, alumino magnesium, and lamp black, azo and diazo dye, a phthalocyanine, and dioxazine, is mentioned. In addition, although it does not dare need an organic solvent in this invention, it does not bar using this.

[0055]

[Embodiment of the Invention] An urethane prepolymer is compounded, and although it can be chosen suitably whether another equipment is processed into the adhesives after migration or both processings to urethane prepolymer composition and adhesives are made to serve a double purpose with one equipment, an operation gestalt is stated by the former approach here.

[0056] In invention of claim 1, in order to improve the adhesive property of the urethane prepolymer of an isocyanate radical end, a N-methyl-2-pyrrolidone is blended.

[0057] After teaching polyol to a reaction container, carrying out reduced pressure dehydration enough and mixing the poly isocyanates, such as superfluous 4, 4'-diphenylmethane diisocyanate, or its crude material, it is made to react on the usual urethane composition conditions (for it to be 4 hours at 80 degrees C). After cooling, a N-methyl-2-pyrrolidone is blended and adhesives are obtained. At this time, other modifiers (specifically a calcium carbonate, dioctyl phthalate, the end of superfines silica etc.) without the effect of moisture may be blended.

[0058] In invention of claim 2, the blending ratio of coal of the N-methyl-2-pyrrolidone to the urethane prepolymer of a desirable isocyanate radical end for considering as a high adhesive property by hypoviscosity further is 0.1 - 30 mass section to the prepolymer 100 mass section in invention of claim 1.

[0059] As a gestalt of implementation of invention of claim 3 and claim 4, it is based on below. Although each may be used independently, since the prepolymer which the direction used together excelled [hypoviscosity] in the adhesive property is obtained, polyether polyol and polyester polyol are explained using mixed polyol. After choosing more than a kind of polyoxypropylene polyol and the polyol of a hydroxyl-group end which carried out the esterification reaction of the ethylene glycol to the adipic acid as polyester polyol as polyether polyol, teaching a reactor and carrying out reduced pressure dehydration enough, the poly isocyanates, such as superfluous 4, 4'-diphenylmethane diisocyanate, or its crude material, are added, and it is made to react on the usual urethane composition conditions (for it to

be 4 hours at 80 degrees C). After cooling, a N-methyl-2-pyrrolidone is blended and adhesives are obtained. At this time, other modifiers (specifically a calcium carbonate, dioctyl phthalate, the end of superfines silica etc.) without the effect of moisture may be blended.

[0060] Furthermore, in order to make into fast curability further the adhesives obtained above, the urethane curing catalyst chosen from the screw (morpholino ethyl) ether, the screw (2, 6-dimethyl morpholino ethyl) ether, and the screw (3, 5-dimethyl morpholino ethyl) ether may be blended. [0061] Thus, the obtained adhesives are contained under a nitrogen air current in containers which can be sealed, such as a cartridge and a pail can. After receipt is pressed out, takes out adhesives by approaches, such as spatula picking, and applies them to adherend. The method of application shall be suitably chosen according to adhesion work habits. Usually, it applies using a comb plate etc. [0062]

[Example] Hereafter, in order to show this invention still more concretely, an example is described based on the operation gestalt of invention.

[0063] To 1L separable flask, the 2 organic-functions polypropylene-glycol (trade-name bamboo rack P-21: Takeda Chemical Industries, Ltd. make) 300 mass section of molecular weight 2000, (Example 1) The 3 organic-functions polypropylene-glycol (trade name bamboo rack P-31, Takeda Chemical Industries, Ltd. make) 100 mass section of molecular weight 3000, The 2 organic-functions polyester system polyol (trade name ADEKANYU-ace F7-67, Asahi Denka Kogyo K.K. make) 100 mass section of molecular weight 2000 is taught. When it cooled to the room temperature and solution temperature fell at 50 degrees C after carrying out reduced pressure dehydration under 1-hour churning at 100 degrees C, added the KURUDO MDI(trade name Sumi Joule 44V-20, Sumitomo BAIERUN urethane company make) 250 mass section, it was made to react at 80 degrees C for 4 hours, and the urethane prepolymer was obtained. This prepolymer 750 mass section was moved to 5L planetary mixer, and the fully dried whiting (trade name NS# 2300, the Japanese east powdering industrial company make) 500 mass section, the impalpable powder silica (trade name RY200, product made from Japanese Aerosil) 50 mass section, and the DOP200 mass section were added, and it fully mixed. The N 1 MECHIRU 2-pyrrolidone 75 mass section was added, and 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent of the hypoviscosity which is a non-solvent was obtained.

[0064] (Example 2) Five mass sections combination of the screw (2, 6-dimethyl morpholino ethyl) ether (trade name U-CAT2041, San Apro make) was carried out by the final process of the adhesives of an example 1. In addition, the viscosity value was comparable as the example 1.

[0065] (Example 1 of a comparison) The N-methyl-2-pyrrolidone (NMP) in a final process was not blended with the adhesives of an example 1.

[0066] (Example 2 of a comparison) The example 1 was followed except having used the petroleum system hydrocarbon solvent (trade name shell ZORU 71, product made from shell JAPAN) for the change of NMP.

[0067]

[Table 1]

	実施例 1	実施例 2	比較例 1	比較例 2	比較例 3
PPG(1)	300	300	300	300	300
PPGØ	100	100	100	100	100
ポリエステル①	100	100	100	100	100
クルードMD I	250	250	250	250	250
炭酸カルシウム	500	500	500	500	500
DOP	200	200	200	200	200
<b>微粉末シリカ</b>	50	50	50	50	50
アミン系触媒	_	5	-	_	5
NMP	75	75	-	_	· <b>-</b>
シェルゾール71 -		_	_	75	75

ただし、PPGD:タケラックP-21 PPG②:タケラックP-31

ポリエステル①: アデカニューエース F7-67

アミン触媒:U-CAT2041

[0068] (Example 3 of a comparison) The example 2 was followed except having used the petroleum system hydrocarbon solvent (trade name shell ZORU 71, product made from shell JAPAN) for the change of NMP.

[0069] Combination of examples 1 and 2 and the examples 1, 2, and 3 of a comparison was shown in Table 1.

[0070] Thus, spreading workability (viscosity and viscosity), an adhesive property, and fast curability were evaluated by the following approach about obtained 1 liquid moisture hardening mold urethane system adhesives constituent.

[0071] The viscosity of a constituent and viscosity (it is called the "TI value" a thixotropic index and the following.) estimated spreading workability (viscosity and viscosity), namely, the viscosity (23 degrees C) in rotational frequency 20 r/min and 2 r/min of B mold rotational viscometer -- measuring -- further -- 2 r/min / 20 r/min=TI value was calculated. furthermore, the trowel when actually applying practical spreading workability with a comb plate -- the lightness of \*\*\*\*\* estimated. Evaluation was made into the three-stage of O, \*\*, and x. Here, good and \*\* mean good, x means a failure, and O cannot present practical use with it in x. moreover, the TI value -- the index of the right and wrong of spreading workability -- becoming -- the one where the numeric value is larger -- a trowel -- spreading workability. such as \*\*\*\*\*, expresses a good thing.

[0072] An adhesive property to 22\*\*1 degree C and indoor [ which were adjusted to RH (standard conditions) 55\*\*5% ] 1.5mm in a flexible board with a thickness of 8mm and thickness Width of face of 25mm, a plasticized-polyvinyl-chloride sheet with a die length of 150mm, and the above-mentioned adhesives constituent are left from the previous day. Coverage is about 250 g/m2 with a comb plate. It applied to the flexible board at the filament so that it might become, and the previous vinyl chloride sheet was stretched after the 30-minute passage of time, breakaway bonding strength was measured 90 degrees by speed-of-testing 50 mm/min after care of health for three days, and the bonding strength and the fracture situation at that time were compared. Although the adhesive property to adherend is good in the location of fracture being an adhesives layer, when it comes to the interface of adherend and adhesives, it can be judged that an adhesive property is conversely bad.

[0073] The time amount of until tuck free estimated fast curability.

[0074] That is, time amount (tack free time) until it stops sensing a tuck by applying and carrying out finger touch of the adhesives constituent under the same conditions as adhesive evaluation was found. It is fast curability, so that tack free time is short.

[0075] The evaluation result of spreading workability, an adhesive property, and fast curability was

shown in Table 2.

[0076] The TI value of the example which is the characteristic as which 1 and 2 express the structural viscosity which is hypoviscosity and is the viscosity ratio of 2 r/min and 20 r/min from the result of Table 2 was large, and it was good. [ of the spreading workability in a comb plate ] the example 1 of a comparison -- very much -- hyperviscosity -- becoming -- a trowel -- \*\*\*\*\* was also very heavy and it was not what is equal to spreading practically. Moreover, although hypoviscosity was obtained and the comparatively large TI value was acquired in the examples 2 and 3 of a comparison, adhesive properties ran short far and tuck tally time amount especially also became long in the example 2 of a comparison. [0077]

[Table 2]

Š	010 2						
			実施例 1	実施例 2	比較例 1	比較例 2	比較例 3
<b>逸布作業性</b>	粘度 Pa·s	2r/min	225	252	320	116	152
		20r/min	44 .	48	145	33	38
	TI值		5.1	5.3	2,2	3,5	4.0
	コテさばき		0	0	×	0	Ó
接着性	90度はく離接着強さ N/m		2300	2500	1200	600	1000
	破断の位置		A100	A 100	AF 100	AF 100	AF100
タックフリー時間 :分			50	30	80	90	50

※ 破断の位置

A 100: 接着剤層の破壊が100%

AF100: 接着剤層と被着体との界面破壊が100%

[0078] In the adhesive result, although the bonding strength of the abbreviation one half came out in the high example 1 of a comparison of viscosity to the bonding strength exceeding 2000 N/m having discovered each example, in the examples 2 and 3 of a comparison, further, bonding strength became low, and the location of the fracture also became the interface of a plasticized-polyvinyl-chloride sheet and a flexible board, and it brought a scarce result at the adhesive property.

[0079] The time amount of until tuck [ which saw fast curability ] free showed the fast curability which is not inferior even if it excels a solvent type in an example 30 - 50 minutes, and early.

[0080] On the other hand, in the example 1 of a comparison, and the example 2 of a comparison, until tuck free took 80 - 90 minutes, and it was inferior to fast curability. Moreover, in the example 3 of a comparison, although time amount until it becomes a tuck free-lancer according to the effectiveness of a catalyst was shortened, the good adhesive property was not acquired like other examples of a comparison.

[0081]

[Effect of the Invention] 1 liquid moisture hardening mold urethane system liquefied type adhesives constituent of this invention can be used as non-solvent form adhesives which do not contain the organic solvent of a low-boiling point thru/or the inside boiling point as explained above. The adhesives constituent of hypoviscosity is obtained compared with 1 liquid moisture hardening mold urethane system adhesives of the conventional non-solvent form, and since the structural viscosity excellent in spreading workability, such as a comb plate in a room temperature, is shown, spreading can be performed easily. About adhesion, fast curability is provided, and bonding strength is good and demonstrates the adhesive property which was especially excellent in the charge of difficulty binders, such as a metal, rubber, and plastics.

[Translation done.]